

## PATENT SPECIFICATION

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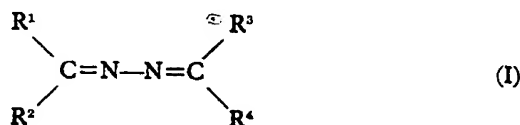
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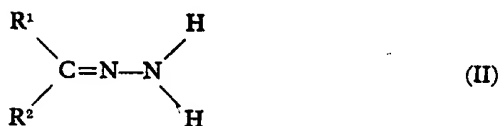
## (54) PROCESS FOR PREPARING HYDRAZINE HYDRATE

(71) We, OTSUKA KAGAKU YAKUHIIN KABUSHIKI KAISHA, a Japanese corporate body, of 10, Bungomachi, Higashi-ku, Osaka-shi, Japan, do hereby declare the invention, for which we pray that a patent may be granted to us, and the method by which it is to be performed, to be particularly described in and by the following statement:—

This invention relates to a process for preparing hydrazine hydrate by hydrolyzing a ketazine of the formula

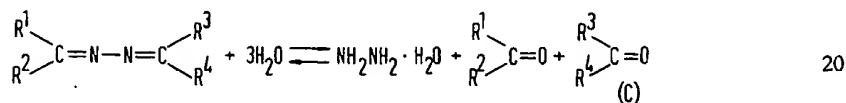
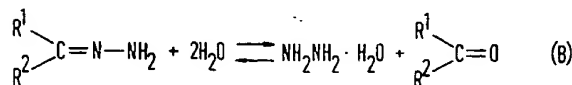
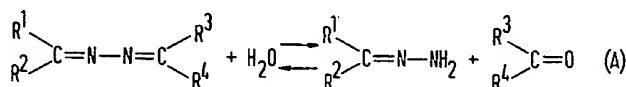


or a hydrazone of the formula



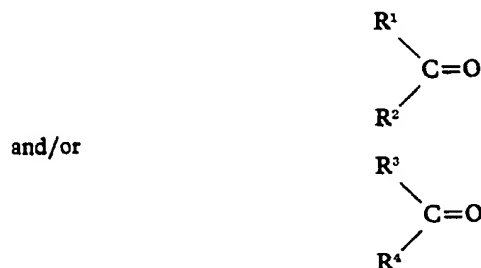
wherein  $R^1$  and  $R^2$  are the same or different and are each alkyl having 1 to 3 carbon atoms, the total number of carbon atoms of  $R^1$  and  $R^2$  being 4 or less than 4,  $R^3$  and  $R^4$  are the same or different and are each alkyl having 1 to 3 carbon atoms, the total number of carbon atoms of  $R^3$  and  $R^4$  being 4 or less than 4.

The hydrolysis of these ketazines and hydrazones may be represented by the following Equations (A) and (B), or by Equation (C) which is a combination of Equations (A) and (B).



The hydrolysis of ketazines and hydrazones (hereinafter referred to simply as "ketazine" unless otherwise indicated) represented by the formulae (I) and (II) will be discussed in detail. First, ketazine undergoes partial hydrolysis in the presence of

water, as shown in Equations (A), (B) and (C) to reach equilibrium (hereinafter referred to as "Equilibrium-1"). Since this hydrolysis reaction is a reversible reaction, the reaction system in which equilibrium is established conjointly contains ketazine, hydrazone, hydrazine hydrate, ketone (free ketone resulting from the reaction and ketone serving as a solvent when ketazine is used dissolved in ketone) and water. Accordingly when the free ketone represented by



is subsequently withdrawn from the reaction system, the tendency for the system to attain equilibrium permits further hydrolysis of ketazine. Thus, the reaction proceeds to the right to again establish equilibrium (hereinafter referred to as "Equilibrium-2"). When the mole ratio of ketone to hydrazine hydrate in the hydrolysis is defined as

$$K = \frac{\text{Total ketone}}{\text{Total hydrazine}} \quad (\text{D})$$

wherein

$$\begin{aligned} \text{total ketone} &= \left[ \begin{array}{l} \text{number of moles} \\ \text{of free ketone} \\ \text{in equilibrium} \\ \text{system} \end{array} \right] + \left[ \begin{array}{l} \text{number of moles of} \\ \text{ketone to be formed} \\ \text{by complete hydrolysis} \\ \text{of ketazine or} \\ \text{hydrazone} \end{array} \right] \\ \text{total hydrazine} &= \left[ \begin{array}{l} \text{number of moles} \\ \text{of free hydrazine} \\ \text{in equilibrium} \\ \text{system} \end{array} \right] + \left[ \begin{array}{l} \text{number of moles of} \\ \text{hydrazine to be formed} \\ \text{by complete hydrolysis} \\ \text{of ketazine or} \\ \text{hydrazone} \end{array} \right] \end{aligned}$$

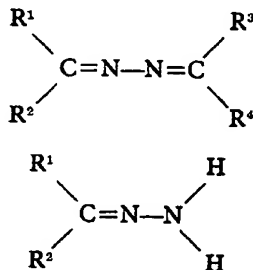
it is theoretically apparent that the reaction system can be rapidly brought to the states of Equilibrium-1 and Equilibrium-2 by reducing the mole ratio. Although the mole ratio appears to be easily reducible by the removal of free ketone from the system by fractional distillation, when this is actually attempted large quantities of water, ketazine and hydrazine are removed together with the ketone, because at Equilibrium-1 the mole fraction ratio of ketone produced is very low, whereas those of water, ketazine and hydrazone are high. Accordingly, there is a problem in how to effectively utilize the heat of vaporization of the water, ketazine and hydrazone to thereby achieve an improved hydrolysis efficiency.

Yamaguchi, Kume *et al.* have already provided a process for preparing hydrazine hydrate by hydrolyzing ketazine using a column having a reflux condenser mounted thereabove and consisting of multiple stages in which ketazine charged in from an upper portion thereof is passed downward through the stages one after another while supplying steam to the column from below the stages (U.S. Patent 3,481,701). The process, accomplished on an experimental scale, achieves a relatively good thermal efficiency, but there is still room for improvement. The process has the drawback that operation in an enlarged apparatus results in a greatly reduced hydrolysis efficiency, rendering the product costly.

We have now devised a process for preparing hydrazine hydrate from ketazine or hydrazone, which process can be operated at a high thermal efficiency and on an industrial scale with a high hydrolysis efficiency.

According to the invention, there is provided a process for preparing hydrazine hydrate by hydrolyzing a ketazine or hydrazine of the formula

or



wherein R<sup>1</sup> and R<sup>2</sup> are the same or different and are each alkyl having 1 to 3 carbon atoms, the total number of carbon atoms of R<sup>1</sup> and R<sup>2</sup> being 4 or less than 4, R<sup>3</sup> and R<sup>4</sup> are the same or different and are each alkyl having 1 to 3 carbon atoms, the total number of carbon atoms of R<sup>3</sup> and R<sup>4</sup> being 4 or less than 4, which process comprises the steps of:

- (1) charging the ketazine or hydrazone in the form of an aqueous or ketonic solution into a fractional distillation column wherein free ketone is removed from the solution, supplying the residual solution to the first hydrolysis reactor of a series of at least two hydrolysis reactors, passing the residual solution in each reactor to the successive reactor to hydrolyze the solution, each of the reactors having a contact column on the top of the reactor, supplying the residual solution from the final reactor in the series of reactors to an upper portion of a fractional distillation column to recover hydrazine hydrate as an end product,
- (2) charging vapour produced in the hydrazine hydrate recovering fractional distillation column to the lower portion of the contact column of the final reactor to bring the vapor into countercurrent contact with a portion of the reaction solution from the same reactor flushed into the upper portion of the contact column, and charging the vapor discharged from the reactor to the preceding reactor to similarly bring the vapor into countercurrent contact with a portion of reaction solution flushed from said preceding reactor,
- (3) supplying the vapor discharged from the first reactor to the first-mentioned fractional distillation column to recover ketone formed by the hydrolysis of reaction solution from the vapor along with free ketone from the starting solution, and
- (4) causing a solution to remain in each of the hydrolysis reactors in an amount not smaller than the amount of aqueous solution of hydrazine hydrate recovered from the hydrazine hydrate recovering means per hour, the solution remaining in the hydrolysis reactors having a mole ratio of 2.0 to 1.2 which mole ratio is defined by K of the following equation:

$$K = \frac{\text{Total ketone}}{\text{Total hydrazine}}$$

wherein

$$\begin{aligned}
 \text{total ketone} &= \left[ \begin{array}{c} \text{number of moles} \\ \text{of free ketone} \\ \text{in equilibrium} \\ \text{system} \end{array} \right] + \left[ \begin{array}{c} \text{number of moles of} \\ \text{ketone to be formed} \\ \text{by complete hydrolysis} \\ \text{of ketazine or} \\ \text{hydrazone} \end{array} \right] \\
 \text{total hydrazine} &= \left[ \begin{array}{c} \text{number of moles} \\ \text{of free hydrazine} \\ \text{in equilibrium} \\ \text{system} \end{array} \right] + \left[ \begin{array}{c} \text{number of moles of} \\ \text{hydrazine to be formed} \\ \text{by complete hydrolysis} \\ \text{of ketazine or} \\ \text{hydrazone} \end{array} \right]
 \end{aligned}$$

Our research has revealed that the behaviour of the solution having the above-defined K value in the range of 2.0 to 1.2 materially influences the efficiency of

hydrolysis in the production of hydrazine hydrate from ketazine. First, because the solution having a K value of 2.0 to 1.2 reaches Equilibrium-1 at a low velocity, the hydrolysis efficiency can be remarkably improved by causing the solution having such K value to remain in the hydrolysis reactor in an amount larger than the amount of final reaction product to be withdrawn from the hydrazine hydrate recovering means per hour. Second, the reduction in the linear velocity of evaporation of the solution having a K value of 2.0 to 1.2 has been found to increase the hydrolysis efficiency of the solution. In the present invention, therefore, part of the solution drawn off from a low portion of the hydrolysis reactor is flushed into an upper portion of a contact column on the top of the reactor.

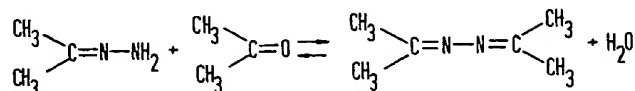
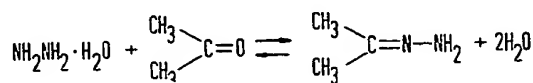
In order that the invention may be more fully understood, reference is made to the accompanying drawings, in which:

Fig. 1 is a graph showing the influence of the amount of reaction solution retained in the hydrolysis reactor on the efficiency of hydrolysis;

Fig. 2 is a graph showing the influence of linear velocity of evaporation of the reaction solution in the hydrolysis reactor on hydrolysis; and;

Fig. 3 is a flow chart showing a mode of practicing this invention.

Suitable amounts of acetone are added to a 10 W/V% aqueous solution of hydrazine to prepare solutions whose mole ratios, i.e. K values, are 0.5, 1.0, 1.5, 2.0 and 2.25 respectively. As will be apparent from Equations (A) and (B), the following equilibrium is established in these solutions.



Subsequently a 200-ml portion and a 500-ml portion of the solution having the above-specified mole ratio are placed respectively into 200-ml and 500-ml distilling flasks having the same evaporation surface area. Each of the solutions is heated for 10 minutes to obtain 10 ml of fraction. The mole ratio K of the fraction is determined. The results are shown in Fig. 1.

Fig. 1 indicates that the composition of the fraction, in other words the composition of the residue, depends largely on the amount of the solution. More specifically with original solutions ranging from 1.2 to 2.0 in the value K, the mole ratio of the fraction rises or the mole ratio of the residue lowers with the increase in the amount of the solution. This shows that the increase in the amount of the solution greatly promotes the hydrolysis reaction. Thus higher hydrolysis efficiencies are advantageously attainable when the reaction solution having a K value of 2.0 to 1.2 is made to remain in the system in a larger quantity than a reaction solution whose K value is below 1.2. In the present invention, therefore, the volume of reaction solution having a K value of 2.0 to 1.2 and retained in each hydrolysis reactor is preferably 2 to 10 times the volume of hydrazine hydrate recovered per hour in view of the overall scale of the apparatus and yield.

The same procedure as above is followed to prepare solutions whose mole ratios, i.e. K values, are 0.5, 1.0, 1.5, 2.0 and 2.25. Two 350-ml portions of the solution are placed respectively into distilling flasks having different evaporation surface areas, namely 200 cm<sup>2</sup> and 50 cm<sup>2</sup>. Each of the solutions is heated for 10 minutes to obtain 10 ml of fraction. The mole ratio K of the fraction is determined. Fig. 2 shows the results, which reveal that under constant heating conditions, namely with a constant amount of evaporation, the evaporation surface area profoundly influences the composition of the fraction, in other words the composition of the residue, where the K value is in the range of 2.0 to 1.2. More specifically stated, with reaction solutions ranging from 1.2 to 2.0 in mole ratio K, the mole ratio of the fraction significantly increases or the mole ratio of the residue lowers with the increase in the evaporation surface area, namely with the decrease in the linear velocity of evaporation. By "linear velocity of evaporation" we mean

## Amount of evaporation

## Evaporation surface area

Manifestly, an increased evaporation surface area or reduced linear velocity of evaporation leads to a higher hydrolysis efficiency. According to the present invention, therefore, part of the reaction solution drawn off from the hydrolysis reactor is circulated thereto in the form of a flush into the vapor phase in the upper portion of the reactor to form a three-dimensional evaporation surface for the purpose of remarkably enlarging the evaporation surface area or of greatly reducing the linear evaporation velocity. In view of the amount of reaction solution retained in the reactor and other conditions, the linear velocity of evaporation according to this invention is usually not higher than 10 cm/sec., preferably 0.1 to 0.6 cm/sec.

This invention will be described below in greater detail with reference to the flow chart of Figure 3 showing the process of this invention for illustrative purpose only. Ketazine or hydrazone starting material, is fed to a fractional distillation column which consists of a distillation still 2 having a steam heating means 3 and a fractionating column 2' of a packed column type provided on the top of the distillation still 2. The starting material is charged usually in the form of an aqueous solution having a mole ratio K of 2 or less, or of a ketonic solution having a mole ratio K of greater than 2 as dissolved in acetone or methyl ethyl ketone. The aqueous or ketonic solution of the starting material is charged to the fractionating column 2' through a feed line 1. When desired, the solution is heated in the distillation still 2 by the steam heating means 3, and the ketone fractionated from the starting material is led from the column 2' through a line 28 to a condenser 29, from which it is recovered by way of lines 30 and 31.

Preferably, the ketone is recovered from the starting material in the fractional distillation column to the greatest possible extent to lower the mole ratio K of the material as much as possible. The residual solution is drawn off from the distillation still 2 via a line 4 and sent to a first hydrolysis reactor 6 by a pump 5.

When the starting material is a ketonic solution of ketazine, water is supplied to the reactor 6 through a line 33. A portion of the reaction solution in the reactor 6 is drawn off from its bottom via a line 7, forced by a pump 8 through a line 9 and flushed into a contact column 6' on the top of the reactor 6, whereupon the solution comes into contact with vapor from a contact column 11' on the top of a second hydrolysis reactor 11 to be described later. Consequently free ketone in the solution which has been in equilibrium within the reactor 6 is transferred into the vapor. This materially increases the ketone concentration of vapor from the contact column 6'. The ketone-containing vapor is therefore led via a line 27 and a flow control means P to the fractionating column 2', from which the ketone is recovered along with the free ketone from the starting material. The water, ketazine and hydrazine in the vapor within the contact column 6' are circulated downward into the reactor 6, in which the heat of the vapor is effectively utilized. The reaction mixture having a reduced K value is forced by the pump 8 through lines 7 and 10 to the second hydrolysis reactor 11, in which it is similarly subjected to hydrolysis. A portion of the reaction solution in the reactor 11 is drawn off from its bottom via a line 12, forced by a pump 13 through a line 14 and flushed into the contact column 11' whereupon the solution comes into contact with vapor from a contact column 16' on the top of a third hydrolysis reactor 16. The residual solution from the second reactor 11 is sent by a pump 13 through lines 12 and 15 to the third hydrolysis reactor 16, in which it is subjected to hydrolysis. Similarly as described above, a portion of the reaction solution from the reactor 16 is flushed into the contact column 16' by a pump 18 through lines 17 and 19. In this way, the mole ratio K of the solution is progressively lowered. The residual solution obtained from the third reactor 16, although still containing ketazine, has a high hydrazine concentration. It is in the form of an aqueous solution of hydrazine hydrate having a mole ratio for example of about 1.2. The aqueous solution is sent by the pump 18 through lines 17 and 20 to an upper portion of a fractional distillation column 21. The column 21 is equipped with suitable heating means such as steam heating means 22. The vapor given off from the reaction solution within the fractional distillation column 21 comes into countercurrent contact with the aqueous solution charged into the upper portion thereof via the line 20. The ketone and ketazine fractionated from the solution and vapor are led through a line 24 into the contact column 16'. Similarly the vapor from the contact column 16' is forwarded through a line 25 to the contact column 11', from which the vapor is conducted through a line 26 to the contact column 6'. The vapor from the contact column 6' is sent through the

line 27 and flow control means P to the fractionating column 2'. Thus the vapor is effectively utilized as a heat source for the reactor and ketone recovering fractionating column. On the other hand, an aqueous solution of hydrazine hydrate is continuously recovered from the recovery column 21 via a line 23. A portion of the ketone recovered from the fractionating column 2' by way of the line 28, condenser 29 and line 30 can be refluxed through a line 32 to the fractionating column 2', in which the ketone may be contacted with the vapor from the ketone recovering means 2 and from the contact column 6' for the recovery of ketazine, hydrazine hydrate and water contained in the vapor.

Because the reaction mixture can be hydrolyzed with greatly improved efficiency when it has a mole ratio K of 2.0 to 1.2 as already described, it is essential that a reaction mixture having a K value within this range be retained in the reactors 6, 11 and 16 and that the amount of the mixture retained in each of the reactors be not smaller than the amount of aqueous hydrazine hydrate recovered per hour.

It will be apparent from the foregoing description that in the process of this invention, the thermal energy supplied to the hydrazine hydrate can be effectively utilized in the reactors and ketone recovering means in succession, so that the ketone recovering means may be heated to such extent that ketone will be fractionated and ketazine or hydrazone and water are recoverable from the vapor from the contact column 6'.

While the flow chart of Fig. 3 shows three hydrolysis reactors, the number of the reactors is determined in accordance with the scale of the overall hydrolysis system (i.e. the desired yield of hydrazine hydrate), the size of the reactor, the temperature and pressure of hydrolysis reaction, etc. At least two, preferably 2 to 4 reactors are used. The process of this invention may be practiced at atmospheric or elevated pressure. Particularly when the reaction is conducted at an increased pressure, the number of hydrolysis reactors and steam requirement are reducible. Under excessively high pressure, however, hydrazine tends to decompose; the pressure is therefore limited to a maximum of 4 kg/cm<sup>2</sup> gauge.

The temperature of the reaction mixture in the hydrolysis system is also widely variable with the kind of the starting material, K value, reaction pressure, etc. The temperature of the solution in the ketone recovering means is usually 56.5 to 102°C, preferably 85 to 98°C under atmospheric pressure and up to 150°C under elevated pressure. The temperature of the solution in the hydrazine hydrate recovery column is usually 96 to 115°C, preferably 102 to 106°C under atmospheric pressure and up to 160°C under elevated pressure. The hydrolysis in the reactors is conducted at temperatures intermediate of the above-mentioned temperatures and raised progressively from reactor to reactor.

The process of this invention will be described below more specifically, by way of illustration only, with reference to the following Examples.

#### Example 1

To the apparatus shown in Fig. 3 is fed a 15 W/V % aqueous solution of acetone azine at a rate of 90 liter/hr. to continuously produce a 8.08 W/V % aqueous solution of hydrazine hydrate containing 0.5 W/V % of acetone at a rate of 73.4 litre/hr. The acetone is recovered at a rate of 13.6 kg/hr. In each of the hydrolysis reactors 6, 11 and 16, 150 liter of the reaction mixture is retained, whilst the reaction mixture is flushed into each of the contact columns 6', 11' and 16' at a rate of 2,500 liter/hr. The reaction system is maintained at temperatures of 87°C, 95°C, 97°C, 99°C and 105°C in the ketone recovering means 2, reactors 6, 11 and 16, and hydrazine hydrate recovery column 21 respectively.

Based on the theoretical values, 99.7% of hydrazine hydrate and 99.8% of acetone are recovered. Steam consumption is 6.0 kg per kilogram of hydrazine hydrate.

#### Example 2

To the apparatus shown in Fig. 3 is fed a 30 W/V % aqueous solution of acetone azine at a rate of 50 litre/hr. to continuously produce a 20.1 W/V % of aqueous solution of hydrazine hydrate containing 0.85 W/V % of acetone, while recovering the acetone at a rate of 15.3 kg/hr. A 150 liter quantity of the reaction solution is retained in each of the hydrolysis reactors 6, 11 and 16, whilst the reaction solution is flushed into each of the contact columns 6', 11' and 16' at a rate of 2,500 liter/hr. The reaction system is maintained at temperatures of 88°C, 96°C, 98°C, 99°C and 106°C in the ketone recovering means 2, reactors 6, 11 and 16, and hydrazine hydrate recovery column 21 respectively.

Based on the theoretical values, 99.7% of hydrazine hydrate and 99.8% of acetone are recovered. Steam consumption is 7.2 kg per kilogram of hydrazine hydrate.

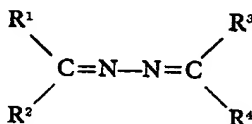
### Example 3

In an apparatus similar to that shown in Fig. 3 except that it is provided with only two hydrolysis reactors 6 and 11, a pressure control valve P is mounted on the line 27 extending from the contact column 6' to the ketone-fractionating column 2' to maintain the system from the contact column 6' to the hydrazine hydrate recovery column 21 at pressure of 3.5 kg/cm<sup>2</sup> gauge. In this state, a 15 W/V % methyl ethyl ketone solution of methyl ethyl ketone azine is fed to the hydrolysis system at a rate of 107 liter/hr. Similarly water is supplied at a rate of 51.4 liter/hr. A 12.5 W/V % aqueous solution of hydrazine hydrate is continuously produced at a rate of 45.6 liter/hr. while the methyl ethyl ketone is being recovered at a rate of 91 kg/hr. A 150 liter quantity of the solution is retained in each of the hydrolysis reactors 6 and 11, whilst the solution is flushed into each of the contact columns 6' and 11' at a rate of 3,000 liter/hr. The system is maintained at temperatures of 96°C, 140°C, 142°C and 151°C in the ketone recovering means 2, reactors 6 and 11, and hydrazine hydrate recovery column 21 respectively.

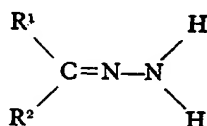
Based on the theoretical values, 99.4% of hydrazine hydrate and 99.6% of methyl ethyl ketone are recovered. Steam consumption is 7.5 kg per kilogram of hydrazine hydrate.

### WHAT WE CLAIM IS:—

1. A process for preparing hydrazine hydrate by hydrolyzing a ketazine or hydrazone of the formula



or



wherein R<sup>1</sup> and R<sup>2</sup> are the same or different and are each alkyl having 1 to 3 carbon atoms, the total number of carbon atoms of R<sup>1</sup> and R<sup>2</sup> being 4 or less than 4, R<sup>3</sup> and R<sup>4</sup> are the same or different and are each alkyl having 1 to 3 carbon atoms, the total number of carbon atoms of R<sup>3</sup> and R<sup>4</sup> being 4 or less than 4, which process comprises the steps of:

- (1) charging the ketazine or hydrazone in the form of an aqueous or ketonic solution into a fractional distillation column wherein free ketone is removed from the solution, supplying the residual solution to the first hydrolysis reactor of a series of at least two hydrolysis reactors, passing the residual solution in each reactor to the successive reactor to hydrolyze the solution, each of the reactors having a contact column on the top of the reactor, supplying the residual solution from the final reactor in the series of reactors to an upper portion of a fractional distillation column to recover hydrazine hydrate as an end product,
- (2) charging vapor produced in the hydrazine hydrate recovering fractional distillation column to the lower portion of the contact column of the final reactor to bring the vapor into countercurrent contact with a portion of the reaction solution from the same reactor flushed into the upper portion of the contact column, and charging the vapor discharged from the reactor to the preceding reactor to similarly bring the vapor into counter-current contact with a portion of reaction solution flushed from said preceding reactor,
- (3) supplying the vapor discharged from the first reactor to the first-mentioned fractional distillation column to recover ketone formed by the hydrolysis of reaction solution from the vapor along with free ketone from the starting solution, and
- (4) causing a solution to remain in each of the hydrolysis reactors in an amount

not smaller than the amount of aqueous solution of hydrazine hydrate recovered from the hydrazine hydrate recovering means per hour, the solution remaining in the hydrolysis reactors having a mole ratio of 2.0 to 1.2 which mole ratio is defined by K of the following equation:

$$K = \frac{\text{Total ketone}}{\text{Total hydrazine}}$$

wherein

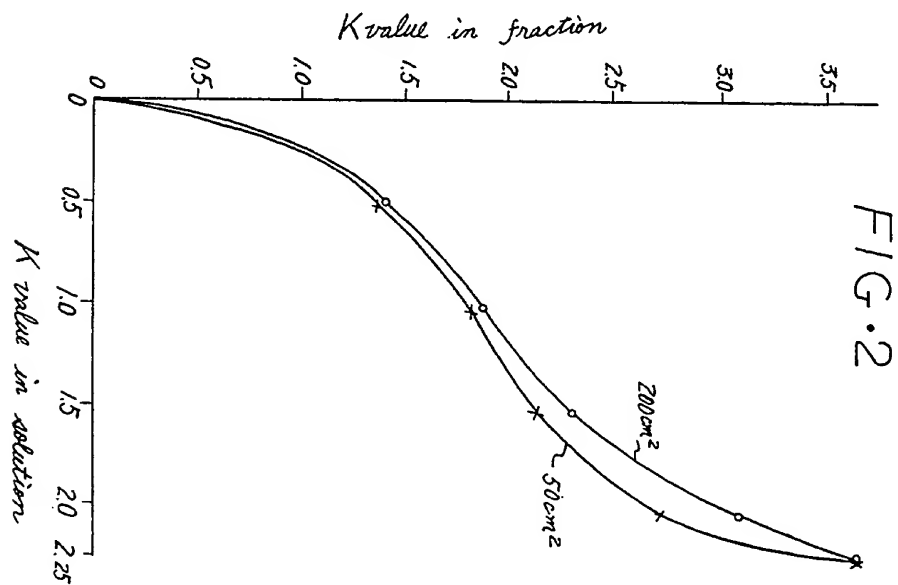
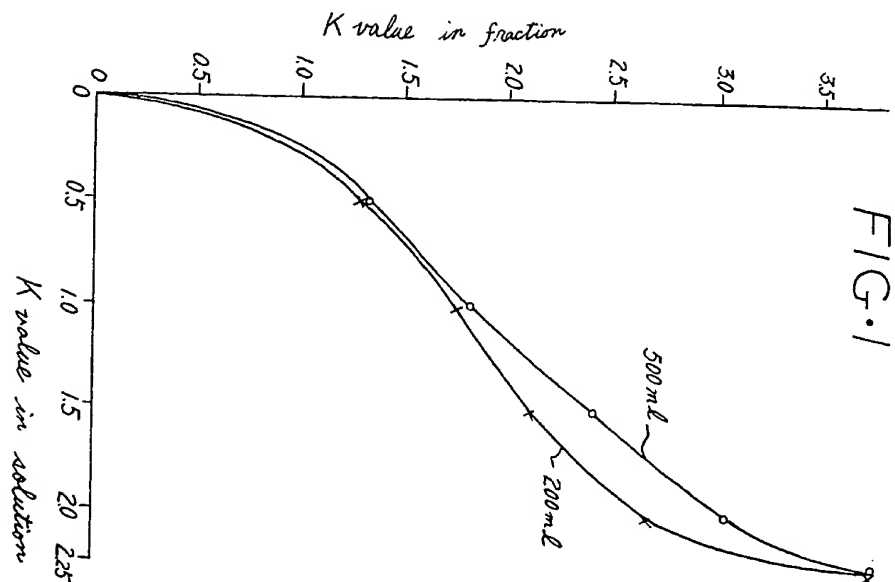
$$\begin{aligned} \text{total ketone} &= \left[ \begin{array}{l} \text{number of moles} \\ \text{of free ketone} \\ \text{in equilibrium} \\ \text{system} \end{array} \right] + \left[ \begin{array}{l} \text{number of moles of} \\ \text{ketone to be formed} \\ \text{by complete hydrolysis} \\ \text{of ketazine or} \\ \text{hydrazone} \end{array} \right] \\ \text{total hydrazine} &= \left[ \begin{array}{l} \text{number of moles} \\ \text{of free hydrazine} \\ \text{in equilibrium} \\ \text{system} \end{array} \right] + \left[ \begin{array}{l} \text{number of moles of} \\ \text{hydrazine to be formed} \\ \text{by complete hydrolysis} \\ \text{of ketazine or} \\ \text{hydrazone} \end{array} \right] \end{aligned}$$

2. A process according to Claim 1 in which said volume of the solution remaining in each of the hydrolysis reactors is 2 to 10 times the volume of aqueous solution of hydrazine hydrate recovered per hour.
3. A process according to Claim 1 in which the amount of reaction solution to be flushed into the contact column from the reactor is such that the linear velocity of evaporation (as herein defined) of reaction solution is not higher than 10 cm/sec.
4. A process according to Claim 3 in which said linear velocity of evaporation is in the range of 0.1 to 0.6 cm/sec.
5. A process according to Claim 1 in which the number of said hydrolysis reactors is 2 to 4.
6. A process according to Claim 1 in which the pressure in the system is atmospheric pressure.
7. A process according to Claim 1 in which the temperature of the solution in ketone recovering means is in the range of 56.5 to 102°C.
8. A process according to Claim 7 in which said temperature is in the range of 85 to 98°C.
9. A process according to Claim 1 in which the pressure in the system is an elevated pressure of up to 4 kg/cm<sup>2</sup>.
10. A process according to Claim 9 in which the temperature of the solution in ketone recovering means is up to 150°C.
11. A process according to Claim 1 in which the temperature of the solution in the hydrazine hydrate recovery column is in the range of 96 to 115°C.
12. A process according to Claim 11 in which said temperature is in the range of 102 to 106°C.
13. A process according to Claim 9 in which the temperature of the solution in the hydrazine hydrate recovery column is up to 160°C.
14. A process according to Claim 1 in which the hydrolysis in the reactors is conducted at temperatures intermediate of those of solutions in the ketone recovery means and hydrazine hydrate recovery column, and raised progressively from reactor to reactor.
15. Hydrazine hydrate prepared by the process of any preceding claim.

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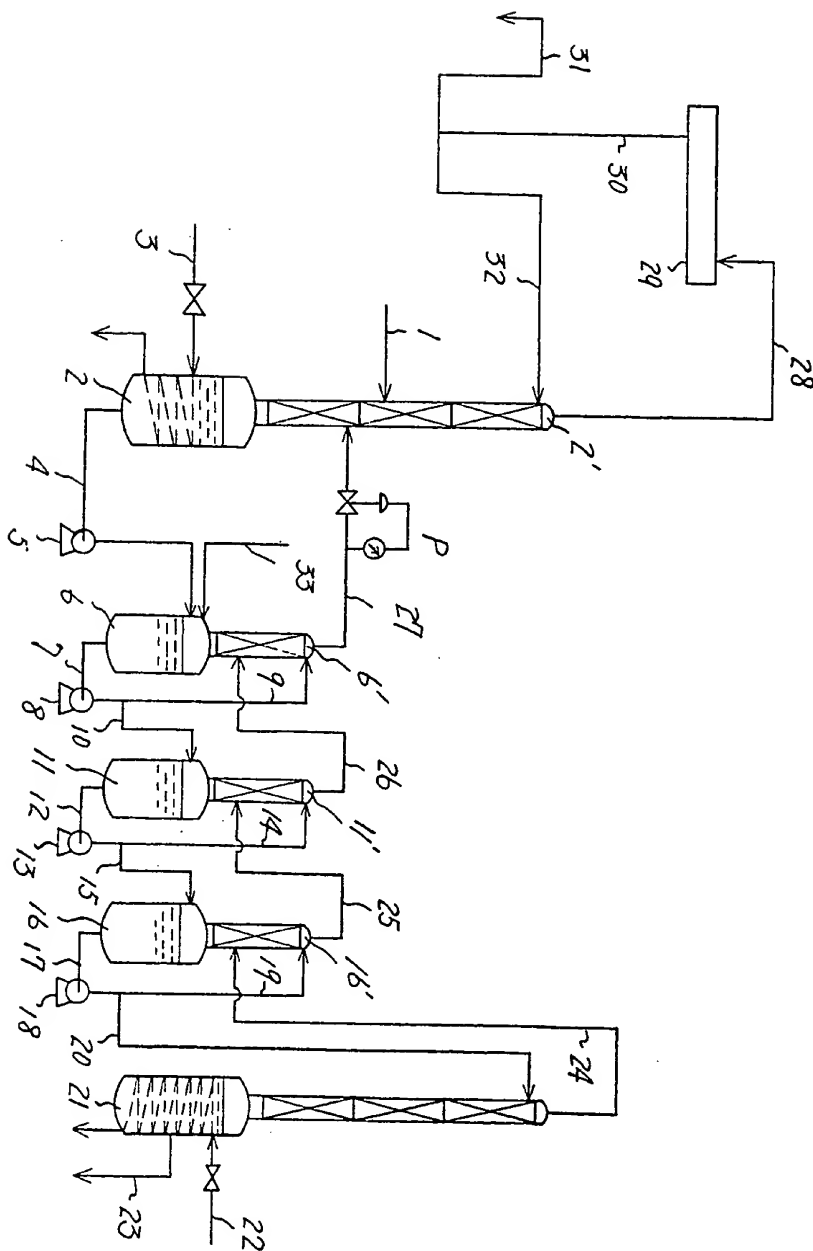


FIG. 3